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INTERFEROMETER HANDBOOK

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Translated  
from the German  
by  
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## 1. Laboratory Interferometer Equipment

### 1. Basic equipment for gas measurement

Laboratory interferometer including 2 stands with columns and base, without chambers  
Four way faucet  
Transformer (220/6V 2.2W with connecting cables)  
Two small lamps F6V 1.8W (as a spares)  
Carry case for the Laboratory interferometer

### 2. Basic equipment for measuring gases and liquids

Basic equipment as given in 1  
Tempering trough and mixer  
Thermometer 0 to 50C, gradation value 0.1 with calibration container for 4 liquid chambers

### 3. Chambers according to use

Two section glass chambers for gas  
100 cm long with receptacle  
50           "  
25           "  
10           "

### 1. Glass liquid chambers for layer thickness

80 mm  
40 mm  
20 mm  
10 mm  
10 mm with insertion for 1 mm layer thickness  
5 mm  
5 mm           "

### 2. Unpacking and setting up of the mechanism

The interferometer is packed in a long carry case with its stand. After the lid has been unscrewed, it is placed next to the case, ready to receive the apparatus, which, only attached to the long tubular body, is first taken out and placed on the two felt covered supports in the lid.

One then joins the sections of the supports (rods and bases) which are located in the bottom and on the side wall of the case, and they are placed at approximately 1 m distance in the center of a 2 in. long work table, and the instrument is carefully placed in the forks. The case is taken away at this point. The ordered gas chambers are to be found in different containers; a container with 4 spaces is anticipated for liquid chambers. It is imperative to conserve not only these containers but also the interferometer carry case.

### 3. Description

#### 3.1. Measurement principle

The appearance of light interference, which is caused by measurement of diversion differences in gases and fluids, results from the following apparatus adjustment:

The light ray stemming from a light source (1), a minute bulb 6V 1.8W, lights up the crack (2) and continues parallelly after filtration through the Kollimator-objective. Light distortion sets in on the edges of the thick double-diaphragm (4) located behind the objective. The lower half of the light ray, which cross the area beside the chamber (5), are led into the distance cone (9) producing in the lens (10) the set lower interference stripe system. The upper section of light rays pass through the double chambers (5) and the compensator plates (6, 11) and also through the distance cone and appear in the lens as the upper, mobile stripe system. Both stripe systems are moved by means of an assistance plate, in a fine horizontal line.

If the light break is very different in the media found in the two chamber halves, a phase displacement sets in in the upper half of the light ray, which depending on the dimension of break variation, results in a complete disappearance (a Fig. 2) or a more limited variation (b) of the upper interference stripe system.

As one changes the slant of the compensator plate (6 Fig. 1) the light path becomes longer or shorter resulting in an equation of the variation of the upper stripe system (c Fig. 2).

The two interference stripe systems are brought to equal positions and similar picture by means of a crank on the compensator plate (6 Fig. 1), which is moved by turning the measuring cylinder (8). The value of the displacement is read in two sections and gives the measurement of break deviation between the test and control material.

The movable compensator plate (6) on a level with an immovable compensator plate (11), brings the interferometric measuring to an exact zero method; the light rays are identical for equal media in both chamber halves, i.e., they reach the view point in a the same swinging phase. The readings on the calculating cylinder are only to be read from this point, the zero point.

#### 3.2. Interferometer

Fig. 3 shows the exterior construction of the laboratory interferometer, consisting of distance tube, tube body with top and Kollimator. The Kollimator protective cap also includes the lighting equipment. The other optical parts of the instrument are strongly built into the interior of the tube body. They have been individually described in the clarification of the measurement principle in connection with Fig. 1.

(5.24.3) (2.14.4)  
The gas chambers and the tempering troughs for liquid chambers, after being placed in the tube, are secured by means of a set screw. These accessories are interchangeable. One may therefore complete a gas-interferometer by adding the necessary sections for liquid tests.<sup>1</sup> An extra adjustment is necessary after addition of a tempering trough.

### 3.3. Test chambers

As the gases and liquids to be measured, very frequently contain acids, the test chambers are only produced out of glass with welded windows. The parallelity of the window plates is therefore established by the most accurate test methods. Because of the great sensitivity of the interferometric measure method and to retain constant work results, it is very necessary constantly to use the chambers, in a position marked by a red dot on the ocular side, while taking great care during insertion into the instrument.

(Details given in 6.1 and 7.1) The position of all chambers has been worked out and attached in a way to be able to make future improvements without having to send the mechanism back.

Gas chambers are available as two section chambers in lengths of 100, 50, 25 and 10 cm. The gas is introduced by means of furnished glass couplings, which are introduced into the chambers by means of conical filed ends, and where possible, are stopped up with paraffin. The attachments for the 100-to-50 cm chambers protrude through gaps in the lid when the tube is closed; they will be connected with the remaining gas ducts after attachment of the four way tap which is provided.

Liquid chambers (Fig. 4) are produced in layer thickness of 80, 40, 20, 10 and 5 mm, and also as 10 mm chambers with a 9 mm insert and 5 mm with 4 mm insert. The tempering trough and mixer and a thermometer are also part of the chambers. The liquid chambers provide 1 cm<sup>3</sup> of test material for every 1 cm of layer thickness. In measurements where only approx. 0.1 cm<sup>3</sup> test material is available the 10 and 5 mm chambers with 9 - 4 inserts are used, from which the 10 mm chamber in Fig. 4 was produced. The layer thickness is changed 1 mm by insert (c) by means of a parallel glass plate in each half of the chamber.

### 3.4. Reading procedure

The reading is taken from the revolving pointer (1 Fig. 5) and the measurement cylinder (2) in two sections. One first determines between which section markings of the revolving pointer the upper rim of the measurement cylinder is located, counts the marks as hundreds (0 to 3000) and to this adds

Note 1) Laboratory interferometers of older models must in this case be sent back to us to have the tempering trough added.

the amount shown on the cylinder with the value of one unit for each mark. The data of both sections (14 and 65 in Fig. 5) therefore together give the reading 1465 cylinder section marks (Teilstrichabstand der Trommel - T. T.).

#### 4. Preparation for use

##### 4.1. Connection of light source

The small lamp 6V 1.8W is connected to a transformer in case of alternating current, which is regulated for 220 or 110V or even for an intermediate current as ordered. A single-pole press or lever switch is suitably installed near the eye-piece connecting into direct current is not permitted by the V.D.E. laws. We recommend procurement of a normal 6-V-accumulator in these rare cases.

##### 4.2. Setting up lighting

The interferometer is shipped from the factory completely adjusted, and it is imperative that this adjustment remain constant under all circumstances. After connecting the lamp, which had been installed before shipment of the instrument, one may quite clearly see the two interference stripe systems - however, more clearly the lower nonmobile system - in the eye-piece. The only adjustment still possible is a quarter twist of the eye-piece to equalize a limited, unclarity in the center of the picture usually only noticeable in cases of old layers. If the light position (5 Fig. 6) has changed in what regards the Kollimator slits, due to transportation, or if a complete readjustment is necessitated because of a lamp change, one may proceed as follows:

With a switched on light one may notice through the eye opening (1) the picture of the light rays (5) projected onto the face of the protection window (7) by the lighting lens (6); this must be perpendicular, parallel to the Kollimator aperture (8) and if possible should appear clearly. This position is easily obtained by turning and pushing the lamp fixture (2) after loosening the screw (3).

The correct aperture lighting and through it a completely clear picture of the interference stripe systems is only to be obtained by guiding an assistant while watching through the eye-piece, in correcting the lamp position by careful displacement and turning of the fixture (2) and setting of the two adjustment screws (4). The two interference stripe systems must be lit up equally and divided by a thin horizontal line; shadows on this line may usually be eliminated by appropriate adjusting of the screws (4) and axial movement of the lamp. The set screw is tightened after achieving the correct position.

(3)

#### 5. General rules of operation

The high sensitivity of the operation and the connected slight measurement border of error, dependent on the chamber length and layer thick-



ness, is of prime importance in interferometer installation.

The greater the chamber length, the more precise the measurement, i.e., the measurement precision increases with the chamber length. On the other hand however, the measurement results are reduced by a larger layer thickness, so that the concentration of the added material of tested substance must not pass beyond a certain limit. Before starting with a measurement scale, one must establish, which chamber contains both the needed precision and the necessary measurement extent. The best chamber length may be calculated according to the formulas given below and the best equipment of the mechanism determined.

Moreover, various rules and advice are to be complied with for all interferometer tests (comparison substance, zero point, measurement process) which are given in detail in Section 5.2 to 5.4.

#### 5.1. Physical basis for chamber length determination

To determine the most appropriate chamber length for the forthcoming tests one must first ask the following questions in case of gas analysis:

- a. What mixture will be tested?
- b. What refractive indices, approximated, do the components have? (The values of the comparison gas  $n_1$  and the added component  $n_2$  are to be taken from charts, for example Landolt-Bornstein or F. Lowe's optical measurements.)
- c. Which % contents  $p_{\max}$  of the added components shall be the highest measured?
- d. Which % contents  $p_{\min}$  of the added components shall be the lowest determined, or which determination is desired?

In every case one will be able to chose the chamber lengths as large as possible so as to achieve the highest possible measurement accuracy. Yet because of the answer to question c we conclude that the chamber can at the most be

$$l_{\max} = \frac{5.5}{p_{\max} (n_2 - n_1)} \text{ mm}$$

long, so as to include the suggested measurement value  $p_{\max} \% (5.5 \cdot 10^{-4} = \text{wave length } (\lambda) \text{ of the light used to calibrate the interferometer, see Section 8.1}).$

On the other hand the answer to d gives the lowest value for a chamber

must be at least

$$l_{\min} = \frac{2 \cdot 10^{-3}}{\rho_{\min} (n_2 - n_1)} \text{ mm}$$

long, when one still desires to know the  $\rho_{\min} \%$  of a material, or when the absolute error of the concentrate measure should at most be  $\rho_{\min} \%$ . (In this end  $5.5 \cdot 10^{-4}$  is replaced with  $2 \cdot 10^{-3}$  for simplification).

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From the advanced formulas we obtain the ratio:  $l = \frac{100 \cdot h \cdot \lambda}{\rho (n_2 - n_1)}$

in which  $h$  stands for the number of interference stripes, which appear in the eye-piece when the chambers are filled.

Example:

Answer to a: An air -  $\text{CO}_2$  - mixture will be tested.

Answer to b: The breaking indices are for air (without  $\text{CO}_2$ ):  $n_1 - 1 = 292 \cdot 10^{-6}$  for  $\text{CO}_2$ :  $n_2 - 1 = 450 \cdot 10^{-6}$

Answer to c: The gas mixture may contain up to 20%  $\text{CO}_2$ .  
In which case the greatest chamber length may be:

$$l_{\max} = \frac{5.5}{20 \cdot 158 \cdot 10^{-6}} = 1.7 \cdot 10^3 \text{ mm} = 170 \text{ cm};$$

one may therefore use the 100 cm; the longest chamber.

Answer to d: 0.03%  $\text{CO}_2$  must still be tested. The shortest chamber length will have to be

$$l_{\min} = \frac{2 \cdot 10^{-3}}{0.03 \cdot 158 \cdot 10^{-6}} = \frac{2 \cdot 10^{-3}}{4.74} = 422 \text{ mm} = 42.2 \text{ cm}$$

one may therefore use the 50 cm chamber.

One could still detect 0.01%  $\text{CO}_2$  in pure air in the 100 cm chamber with a maximum contents of 20%  $\text{CO}_2$ . This reaction sensitivity is different for every type of material. For example, when  $\text{CH}_4$  or  $\text{H}_2$  is measured against air in the 100 cm chamber it is approximately 0.015%, 0.03% with the 50 cm chamber, and approximately 0.06 or 0.15% for the 25 and 10 cm chambers.

The reliability of the measurement (margin of error) and of the measuring range of the chamber length can be seen from the summary below. It is thus assumed that for any given length the same chamber will be used.

Chamber length or layer thickness	Margin of error $n_2 - n_1$	Approximate measuring error
100 cm } for gases	2 . $10^{-6}$	0.00005
50 " }	4 . $10^{-6}$	0.00010
25 " }	8 . $10^{-8}$	0.00020
10 " }	2 . $10^{-7}$	0.00050
50 mm } for liquids	2.5 . $10^{-7}$	0.00063
20 " }	5 . $10^{-7}$	0.00125
10 " }	1 . $10^{-6}$	0.00250
5 " }	2 . $10^{-6}$	0.00500
1 " }	4 . $10^{-6}$	0.01000
	2 . $10^{-5}$	0.05000
Dip-in - refractometer with measurement prism I	2 . $10^{-5}$	0.04000

The margin of error in breakage differences of  $n_2 - n_1$  are given in the second column of the table. This value nearly represents a sectional strip on the measurement cylinder (TT) as approximately 1/20 strip width in the green light used for calibration (Detail in Section 8).

Even small chamber length variations from the nominal measurement must be considered in very precise measurements so as to conserve the needed precision. These variations are given in millimeters with the symbol + or - on the front attachment socket of the gas chamber, indicating whether they are to be added or subtracted from the nominal measure. The exact measurement is directly written on the liquid chambers.

The procedure of choosing the chamber length or the layer thickness is basically the same for liquid chambers as that for gas chambers described above. Where the breakage incidence values for example and comparison liquids are not known, one may establish them approximately before work with the aid of an Abbe- or dip-refractometer.

## 5.2. Choice of the comparison substance

As a general rule it is good to choose the comparison substance from samples with similar light breaking- and dispersion-capacity, as the samples are sectional, mainly in higher concentrations. It should be added, that the section values read off the measuring cylinder will be kept low, as for ex. by this means one may dismiss the reduction to normal pressure and normal temperature during gas mixture measurements. These factors are still to be considered in various elevations, even at mountain stations or near sea level, also in mountain work.

Pure, dry and carbon acid free air serve as comparison gas during gas tests.

Low concentrate solutions, for example tap water is compared to distilled water, river water, against drinking water, etc.

### 5.3. Establishing the zero point

The zero point, the starting point for every interferometric measurement, is that compensator setting by which no phase difference exists between the right and left light bundle. It therefore is unimportant what gas or liquid fills both chamber halves, as long as the same substance, i.e., in this case the comparison substance chosen for the measurement, fills both halves. (See Section 5.2).

Assuming that the instrument is completely set up for the job (compare Section 4, 5 and 5.1 as well as the following points under 6 and 7), the complete interference stripe system appears in the lower viewfield. A wide, light stripe (the zero stripe) may be seen in the center; it is framed by two very narrow, nearly black lines, the "Minima I. order". We also recommend that the appearance of the two next Minima (II. order) be checked, for these stripes must be included in measuring of difficult samples to determine correct installation, for example in the case of weakly colored gases and solutions and in stronger concentrations.

If the measurement drum is rotated until its upper edge is located between the first and second section notch of the rotating indicator<sup>1</sup>, a group of colored stripes appear in the upper previously colorless view area. By turning the cylinder very slowly, a spot will be found where both interference stripe systems correspond completely, the upper being movable while the lower is static.

This procedure is to be followed in all later measurements, so that the cylinder moves in the direction of decreasing numbers, thus eliminating the natural play in the measurement setup. The correspondence of the two interference pictures may only be established when the cylinder turns on one or two stripe widths, bringing the change of the colored frames as closely as possible to the "Minima". The movable picture is then brought back a few stripe widths by turning the cylinder, and is set up as before coming from higher values.

The procedure is repeated and the corresponding data read off every time as shown in Section 3.4<sup>2</sup>. The arithmetic average of these values is the

Note 1) This position was approx. at the 10. section mark, or in the neighborhood of 1000 TT, on some old instruments.

Note 2) Only limited differences (1 to 2 TT) will be encountered after a few exercise tests.

mechanism's 0 point. It holds for the circumstances mentioned in the beginning of this section and is only to be determined in case of complete equalization of temperature and pressure. This circumstance is easily recognized a few minutes after the chamber filling, by the straight, immobile interference stripes.

#### 5.4. Measurement procedure

The measurement always follows directly after establishment of the zero point. This also indicates that the zero point must be newly established for every single measurement - daily for series measurements.

However before removing the comparison substance from the one chamber half, one must ascertain whether the comparison substance or the material to be tested have the greater light breakage; for basically, the substance with higher breakage should be placed in the chamber half closest to the measuring cylinder<sup>1</sup>, therefore on the right hand side in the laboratory-interferometer.

The pressure or temperature equalization is waited for after the filling of the measurement chamber (See Section 6 and 7), with substance. In general only the lower interference stripe system has to be considered, as the upper has moved sideways during the filling of the measurement chamber. The actual measurement process consists of numerically calculating the sum of this movement. This is achieved in the same way as that used to establish the zero point. One must therefore only be careful to have exactly identical interference stripes opposite one another at the moment of reading.

The average of the data on the measurement cylinder, minus the value of zero, is the measurement result, the value of which can be drawn from data given in Section 8.

#### 6. Work method for gas tests

The introductory explanations of the previous sections on interferometric measurement methods had to precede the description of the work method so as to spare the worker mistakes and wasted time. It is indicated to also obviously review these explanations in the following.

##### 6.1. Placing the gas chamber

As mentioned in 3.3, the eye-piece side of the gas chamber is marked by a red point on the adjustment section, which must point to the eye-piece when the chamber is set in place. One takes the chamber with both hands and places it carefully as nearly as possible in the center of the tubular body so that the spring set-screw (5 Fig. 3) automatically catches in the corresponding threaded opening, after placement of the chamber the set-screw is then tightened by light pressure.

Note 1) By this means one always has positive measurement results; otherwise the placing in the eye-piece would only be possible under extremely low breakage differences  $n_2 - n_1$ .

## 6.2. Preparation and filling of a gas test

All strongly colored or dust filled gases are to be eliminated from optical tests by use of the interferometer. Carbon dust is easily arrested by the use of a cotton wool filter. Such filters are also to be recommended in cases where the gas samples are only occasionally dusty, so as to guard the gas chamber window panes against formation of a dust layer through long use, soon causing the interference picture to appear darker. A similar distortion of the interference picture is caused by a coat of water vapor on the chamber windows. As water vapor influences the light breakage of a gas mixture, all gas samples must be dried, by slowly passing the sample through a glass tube containing "Dangel" or  $\text{CaCl}_2$ . The gas sample drying must be undertaken with special care when it has been preserved over water, for example forced into the interferometer by a hydraulic gas meter.

Air from pressure chambers is slowly forced into the chamber after drawing into a small rubber pipe. The measurement chamber is full when the upper interference stripe system stops "wandering". Moreover in all cases where one is working with a certain pressure, one will allow the sample to flow through the chamber, controlling the gas flow by letting the gas escape through a glass dish under water, counting the gas bubbles.

The sample and the comparison gas must definitely have identical pressure for correct measurements. With this in mind, where possible, one allows the open end of the comparison chamber to remain in contact with the air by means of a Natron-chalk or calcium chloride solution. The sample in the chamber may easily be kept at atmospheric pressure by allowing the gas stream passing through the chamber to escape into the room through an open, level capillary of approx. 2 mm interior cross measure of 60 to 80 cm length lying on the table. No error through diffusion can be noticed by this method, even in as light a gas as hydrogen. The gas flow will have to be stopped by a tap during each measurement, so as to be independent of all pressure changes in the approach tube.

Pressure corrections motivated by the ruling air pressure in the immediate vicinity are only of practical importance in cases of obvious divergence from normal atmospheric pressure, for example, in mountain work.

## 6.3. Measurement of flowing gases

Passage of the gas through the chamber is recommended, when the gas to be measured shows extra pressure - be it even very small - to be disposed of. In such cases great value must be placed on correct pre-tempering. This is done by passing the gas through a metal or glass spiral two meters long, which is located in a water container. A tap must be located behind this tempering coil so as to control the gas flow. One may check the working of the tempering setup by testing whether the interference stripe system suddenly assumes its correct position when the tap is closed or whether it only moves towards the spot after a certain period. The latter circum-

stance indicates that the gas was not sufficiently pre-tempered when it entered the chamber. Moreover the direction of motion also gives an answer: warmer gas has a lower light breakage than cold gas. In general streaming gas will not give a completely fixed result, as limited pressure changes always exist. Because of this, the tap between the tempering coil and the gas chamber should be closed before every measurement. A great importance is to be placed on gas purification from dust under this circumstance.

#### 6.4. Specific absorbents for single gases

After a complete publication of the application of the gas-interferometer to industrial hygiene had appeared<sup>1</sup>, the question arose as to the lack of knowledge on solid or fluid absorption media, with which one may isolate one, and only one, ingredient from a mixture, without causing chemical reactions with other ingredients. However, one must assume for such specific absorbents, that the only difference between the contents of the two chamber halves in the gas interferometer consists of the presence of the ingredient to be measured in the one or in the other of the two. 17 gases and vapors with the corresponding absorbent were collected in the table given below from the study on gas analysis by F. Bayer<sup>2</sup>, so as to make these absorption media available to a greater public. We are always grateful for any further completion of the table through practical work.

#### 7. Work method for liquid tests<sup>3</sup>

As there is no appreciable difference between the optical procedure of liquids and gas mixtures the rules given under Section 5 for both states of aggregation are immediately applicable. Therefore, it is only a question of explaining special points of mechanism adjustment and material peculiarities.

##### 7.1. Tempering trough and chamber placement

Laboratory-interferometers which were ordered for gas and liquid tests, are delivered from the factory with a tempering trough built into the tubular body.<sup>4</sup> The eye-piece side of the trough is marked by a red

Note 1) Lowe, F.: *Gewerbehygienische Anwendungen des tragbaren Gasinterferometers in chemisch-technischen Betrieben*. Chem. Ztg. 68 (1944) S. 144 ff.

Note 2) Bayer, F.: *Gasanalyse*. In: *Chemische Analyse*. Bd. 39. 2. Aufl. 1941. Stuttgart: Enke.

Note 3) For interferometric liquid measurements (including titrations) See Fr. Lowe; l.c. p. 271-277.

Note 4) The tempering trough must be adjusted (by changing the four screws with nuts in the floor of the tubular body) in cases of additional orders of accessories for measuring liquids.

Type of Gas	Absorbent	Type of Gas	Absorbent
Ethylene	Bromide solution	Propylene	87% $H_2SO_4$ , which also absorbs butylene
Ammonia	Diluted sulphuric acid	Oxygen	Alkalic oxyhydrochinone solution according to F. Henrich (Cal. Chem. Co. 48. 1915 2006): 100 g KOH filled to 200 $cm^3$ with water, add 10 g triacetyl-oxyhydrochinone unexposed to air oxygen, shake until the solution is clear (comp. Bayers Book, p. 61)
Acetylene	Alkali potassium-iodine-mercury solution 30 g KI + 25 g $HgJ_2$ diluted in 100 $cm^3$ of water, a small piece of KOH is then added to make the solution alkalic		
Hydrocyanic acid	Concentr. $H_2SO_4$	Carbon sulphide	Active carbon
Butylene	87% sulphuric acid, which also absorbs propylene	Hydrogen sulphide	$CuSO_4$ in strong sulfuric acid solution: 200 g concentr. $H_2SO_4$ + 200 $cm^3$ water, + $CuSO_4$ until balanced
Helium	Deeply cooled carbon-A	Nitrous oxide (NO)	a. Freshly neutralized Iron (II) sulfate solution, this however also takes nitric-proxide ( $N_2O$ ) b. Acidified Bromate- or potassium permanganate solution according to Klemenc. Z. anorg. Chem. 122 (1922), 3/5
Iodine in air	Liquid potassium carbonate solution, 2 g $K_2CO_3$ in 15 $cm^3$ of distilled water		
Carbon monoxide	a. Iodine pentoxide <sup>1</sup> b. 125 g copper (I) chloride + 265 g ammonia chloride 750 $cm^3$ water, added in the used surface of a copper spiral		
Carbonic acid	a. Potash lye, 28% by weight b. Hard grainy soda lime		

Note 1) Revealed here (1951) with the kind permission of the gas institute of the Technical College in Karlsruhe.



Type of Gas	Absorbent	Type of Gas	Absorbent
Naphthalene vapor in light gas	Solution of picric acid in water (0.70%) at 0°, cool absorption container in ice water!	Nitrous peroxide	See nitrous oxide under a. and F. Bayer, p. 184ff. a. By deep freezing b. Graining calcium chloride c. Blue silica gel with protective color, regenerative
Ozone	1% KI solution, but O <sub>3</sub> in O <sub>2</sub> provides a stronger solution		

dot, which must point to the eye-piece when installed. After attaching the trough by means of the set-screws (5 Fig. 3), the included thermometer is placed in the left and the handle to activate the stirrer on the right (4 Fig. 4).

The liquid chambers are taken from the already prepared wood retainer, which serves to hold filled chambers during changes and cleaning. To place the chamber in the tempering trough one attaches it to the two twisted pins and watches that the two guide pins underneath of the chamber fixture enter the corresponding openings in the tempering trough by matching the red points.

## 7.2. Preparation and filling of a fluid sample

The tempering trough is filled to the upper edge of the round window with water, as dust free as possible. Keeping the water and the sample at an even temperature is not generally necessary, as the difference of breakage incidence between sample and comparison solution is principally measured in the interferometer. However, pre-tempering of the sample to the temperature of the water in the tempering trough has the advantage of being a great saving in time, as the correct formation of interference pictures is only possible after equalization of the temperature of the sample and of the tempering water. The interference stripes of the upper system cannot be exactly set before this because of their continual movements and distortions. The temperature equalization in the chamber and in the temperature trough - including the measuring process - is considerably accelerated by turning the handle of the stirrer (4 Fig. 4). This procedure also results in displacing the air bubbles suspended on the chamber floor, which disturbs the passage of the lower light pocket.

Use of pipettes with small rubber converters are best adapted for filling of small fluid media. The filling should be conducted as sparingly as possible; it is sufficient to have the windows evenly covered. Certain deposits or cloudiness in the sample may be removed by filtering or centrifuging before the filling, as far as permitted by evaporation hazards.

When measuring non-watery solutions, which usually have a higher breakage index than water, it is recommended to fill the tempering trough with a not easily evaporating solution instead of water, one which has a breakage point nearly equal to that of the sample.

## 7.3. Cleaning of the liquid chambers

The pipettes used to fill the chambers are also well adapted to cleaning. The remaining parts of the sample are taken up with small rolls of filter paper, a few drops of the original solution medium are used to rinse and it is dried as above. The chambers must not be noticeably heated during cleaning; a time loss would otherwise be unavoidable.

Actual cleaning between two tests may completely vanish during series

tests, if one has so much extra substance that one can employ the first filling of the second sample as a cleaning medium. This process is only used during tests of similar samples; it forms the rule for all types of water tests.

One must be certain that no water drops have remained in the tubular body after completion of a series test, as the interferometer must be left at night. The liquid chambers should also be emptied and cleaned out before prolonged work breaks.

## 8. Calibration of the interferometer for measurement evaluation

A calibration table or curve must be established for a practical evaluation of the measurements reached with the interferometer, this should also be done for the instrument and for the studied material. The evaluation then solely consists of readings from these tables.

### 8.1. Instrument calibration

The following intrinsic calibration follows without placement of chambers and without tempering trough, by establishment of the zero value in the white light.

Counting of interference stripes in monochromatic light.

The first work step is conducted according to the instructions in 5.3. The obtained value approx. 30 section strip distances (TT) must be deducted from the measurement values obtained in monochromatic light. One notes it placed in the I calibration table (See below).

The lamp socket with the glow lamp (7 Fig. 3) is pulled out for calibration with monochromatic light and a uniform quicksilver lamp covered with a 5461 Å filter<sup>1</sup> is placed in front of the guardcap (6) opening.

The upper interference strip system is moved one strip at a time by rotating the measurement cylinder during illumination with light of the above wave length, and the corresponding TT-value is read off. This value minus the zero value for white light, gives the number of section values  $t$ , which correspond to the stripe number  $h$  placed in the eyepiece.

Table I			Table II		
$h$	$t$	$\Delta t$	$t$	$h$	$\Delta h$
0	0	30	0	0	0.33
1	30	30	10	0.33	0.34
2	60	31	20	0.67	0.33
3	91		30	1.00	
.	.	.	.	.	.
.	.	.	.	.	.
Zero value for white light: 30 TT.			.	.	.

Note 1) We recovered the monochromatic B filter numbered CZ 32-S45-1).

The results of such a measurement series, are given in the preceding Table (p. 21). Here the first column contains the pre-set stripe value (b), the second column the section value (c) after subtraction of the zero value, and the third column the differentials ( $\Delta t$ ) between two consecutive measurements.

As during measurements only one obtains T-values instead of stripe numbers, from the TT-values after subtraction of the zero point, one must change Table I through calculations, to the point where one can obtain stripe numbers of one to two decimals from the T-values. It follows that Table II, which appropriately passes for 10 to 10T-values, indicates what stripe numbers are connected with these values. Small differences between the values of both Tables which result from rounding off, remain without noticeable influence on the measurement results.

By this calibration the T-values qualified by the mechanism are converted into stripe numbers h which are drawn on the wave length  $\lambda = 5461 \text{ \AA}$ . The importance of the stripe numbers rests on the fact that during measurement of certain materials they are only dependent on the chamber length -filling. One may therefore use them as comparison measurements for various interferometers. In reference to this one also speaks of absolute instrument calibration.

Every interferometer is delivered with a calibration table for the first 20 stripe numbers (approx. 600 TT); it may be completed by the investigator during use, according to preceding remarks, for the total range of approximately 3000TT. The complete absolute-calibration may also be conducted by us when so desired, if this is known to us at the time of the original order.

## 8.2. Calibration for the material to be measured

### 8.21. Empiric calibration

All the above exhaustively treated rules, such as choice of a comparison substance, establishment of the zero point, placing of chambers, etc., are of importance in establishing the calibration curve, from which one may draw the concentration of the measured material for every value on the measurement cylinder.

The necessary tests of known contents are now prepared in the most precise manner possible. This will be easy in most cases of liquids, it is often difficult (See Section 8.22), to produce certain gas concentrates with sufficient precision. W. Kinder deals with this by an appropriate process. The calibration tests are filled and pressured consecutively in the same chamber half (compare Section 5.4, 6.2 and 7.2). The TT-values read on the measurement cylinder are entered in a table with the corresponding concentrations after subtraction of the zero point.

Note 1) Kinder, W.: Production of steam-concentrates in determining breakage and in gas-interferometer calibration. Zeiss-Nachrichter. 2. Sequal (1938) H.7.

One will notice, with certain concentrations of supplementary components, that with the usual installation in the eye-piece, not two but three nearly colorless "Miwina I, orders" are apparent in the upper interference strip system. One is perplexed as to which installation is the correct one, seeing that the difference is a whole strip number, i.e., approx. 30 TT. If one represents the data from individual measurement by a coordinate system, the graph is expressed by a breaking of the straight lines, a so-called "jump". The jump areas are limited by the dispersion characteristics of the substance; measurement errors do not result from this. It is only important to take both readings from the measurement cylinder, to take the TT-value reading every time and to note both values of  $t$  in the table.

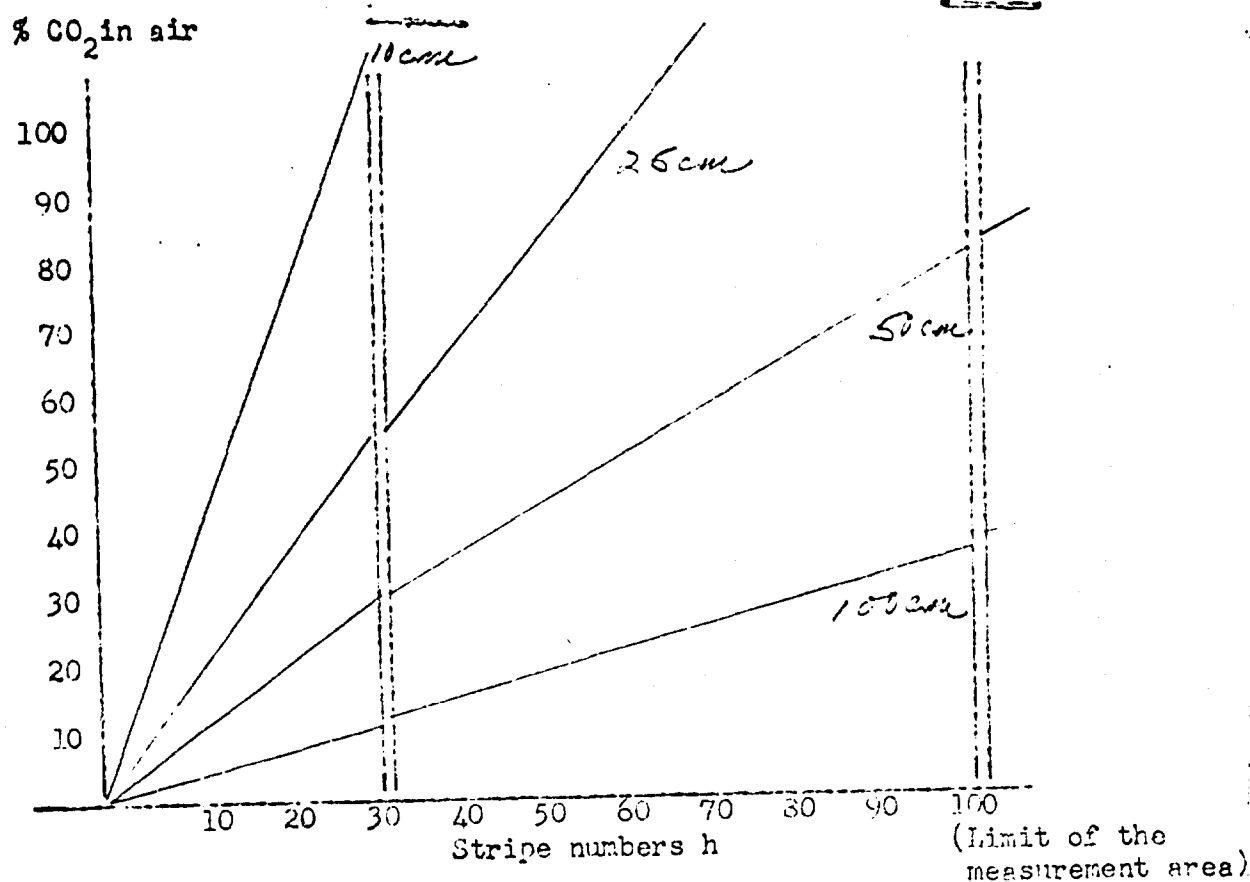


Fig. 7. Calibration curves indicating the CO<sub>2</sub> contents in air for four different chambers

The curves have been entered in Fig. 7 as an example, of how they can result in calibration of a laboratory interferometer with four gas chambers. The same gas mixture, carbon dioxide in dry air, was adopted for all four chambers so as to simplify the process. The T-values are changed into stripe numbers  $h$  by means of calibration Table II, so as to show that a jump area always takes an entire stripe number. The accompanying concentration is also clearly legible in the jump areas, when one considers both possible settings.

It is further to be noted that jumps always appear at the same stripe number independent from chamber length and concentration. The first jump area is located at stripe number 32, the second, already beyond the reach of the measurement screw at approx. 102. The position of this break is different depending on the material measured, a number of jumps, which have nearly equal values, may appear in a calibration curve (for ex. the calibration curve for Benzol in air, as given in the above mentioned work by W. Kinder).

#### 8.22. Calibration through calculation

One is always advised to use calculated calibration when preparation of calibration tests are made impossible by some reason or other. For this one must know the chamber length, the working dispersion of the compensation arrangement in the interferometer, and the breakage force and dispersion of the materials concerned.

As soon as the latter data has been accurately gathered, one is in a position to calculate the calibration tables for changing stripe numbers into concentration percentages for the measured materials against return of the net costs, and to attach this to the mechanism.

In most cases the dispersion values are not yet known with sufficient precision. One may however calculate the calibration tables, but one must watch the position of the jump areas which were arrived at by the measurements with the interferometer and procede with the corresponding corrections in the calculated tables. The calibration curves in Fig. 7 were produced in this manner.

The stripe number  $h$  corresponding to the  $t$  value in calibration Table II is taken for the measurement evaluation, through which the table set up through calculation gives the concentration value of the mixture in question.